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WAFER GUN PROPELLANT

THIOKOL CHEMICAL CORPORATION

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EGLIN AIR FORCE BASE, FLORIDA

Wafer Gun Propellant

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FOREWORD

This program was conducted by the Wasatch Division of Thiokol Chemical Corporation, Brigham City, Utah 84302, under Contract No. F08635-72-C-0137 with the Air Force Armament Laboratory, Eglin Air Force Base, Florida, during the period from 2 March 1972 to 30 June 1973. Mr. Bertram K. Moy (DLDL) managed the program for the Armament Laboratory.

Mr. John A. Peterson, Project Engineer, directed the program for the contractor. He was assisted by Messrs, Raymond M. Price, Russell Reed, Emil A. Lawton, Frank H. Bell, Norman H. Lundstrom, Ronald L. Borcherding, Graham C. Shaw, Michael R. Harper, Leland E. Davis, and Ralph R. Quarles.

This technical report has been reviewed and is approved.

DALE M. DAVIS
Director, Guns and Rockets Division

ABSTRACT

The wafer gun propellant studied in this program is a composite solid propellant with a moldable binder. It contains the cyclic nitramine, HMX, for an oxidizer and has the advantages of high impetus, relatively low flame temperature, and a manufacturing process adaptable to high volume production with good producibility. The wafer gun propellant is punch-pressed from a dry powder into a disc or wafer with nearly neutral burning characteristics. It is then oven cured. The wafer configuration permits an easily varied burning web, since the pressed wafer thickness is a variable controlled by changing pressing dies. The pressed wafer fabrication technique permits a higher oxidizer solids loading than is presently possible with a conventional extruded composite propellant. Production involves fewer process steps than a conventional colloidal gun propellant.

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SECTION I

INTRODUCTION

The rapid fire weapons currently in use have a relatively short barrel life. This short life is a result of high propellant combustion temperatures and high rates of fire. The use of a propellant having a lower combustion temperature should result in a significantly increased barrel life and reduced weapon system cost.

Composite solid propellants, because of their high energy content, adjustable impetus, and flame temperature, have been studied as possible replacements for conventional colloidal gun propellants. Various studies have shown that composite propellants containing cyclic nitramines have a relatively low average flame temperature which should result in increased barrel life.

Composite propellants are usually prepared by mixing a solid oxidizer with a liquid binder; however, in this program a binder composed of all solid ingredients was used. Many compositions are not extrudable without either solvent extension or a reduction in oxidizer solids loading, or both. Therefore, many formulations which cannot be extruded may be pressed into a thin wafer or disc which has a nearly neutral burning surface, though it is slightly regressive.

The resulting wafer gun propellant is attractive for a number of reasons:

A higher solids loading is possible than with typical, extruded composite gun propellants.

Propellant isochoric (constant volume) flame temperature is much lower than that of a colloidal nitrocellulose (NC) propellant of equal mass impetus.

The mean molecular weight of combustion products is much lower than an NC propellant of equal mass impetus.

A mass impetus of over 400,000 ft-lbf/lbm is available at a solids loading of 85.0 weight percent.

Wafer gun propellant fabrication requires fewer process steps than colloidal propellants.

Composite nitramine gun propellants are not without limitations. At an HMX oxidizer weight mean diameter of 2-microns, a burning rate exponent [(n in the equation $\dot{r}_b = A\ (P_{AVG}/10,000)^n$ in./sec)] less than 0.90 has not been demonstrated at solids loadings of 75.0 weight percent and higher without the addition

of a burning rate modifier. Unless a burning rate exponent of less than 0.90 was demonstrated with adequate energy, a tractable cannon propellant was not deemed probable.

This program entailed the research and development of a pressable composite solid propellant formulation containing the cyclic nitramine, HMX, as the oxidizer and a polyethylene glycol/polyurethane binder. This task included theoretical formulation optimization, development and optimization of processing techniques, combustion and hazard studies of the formulated propellant, and the manufacturing of the formulated propellant for Air Force test and evaluation.

SECTION II

PROGRAM OBJECTIVES

This program had two principal objectives:

1. To develop a composite solid gun propellant with HMX as the oxidizer and a solid moldable binder that has the following characteristics:

   a. A mass impetus not less than 360,000 ft-lbf/lbm.*

   b. An isochoric flame temperature not greater than 2,600°K. *

   c. A mean gas molecular weight of combustion products not greater than 20.00 Atomic Mass Units. *

   d. A burning rate exponent** of 0.90 or less.

2. To demonstrate the feasibility of processing propellant wafers with the above characteristics which will have the postulated neutral burning characteristics and satisfactory loading density. The specified dimensions in the study were a 0.250-inch diameter with a thickness of 0.020 inch.

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*P_c = 5,000 psia

**Burning rate exponent defined as n in the equation $r_b = A\ (P_{AVG}/10^4)^n$ in./sec

SECTION III

TECHNICAL APPROACH

A new solid gun propellant can be achieved by inporporating finely ground HMX in a dry binder, pressing the blended propellant into wafers and subsequently curing the wafers in an oven. This is a new approach to increasing propellant energy content. By pressing the propellant rather than extruding it, a higher oxidizer solids loading is possible.

SECTION IV

PROPELLANT

A. WAFER GUN PROPELLANT

The wafer gun propellant investigated during this program was composed of the crystalline nitramine HMX blended with a dry binder system and pressed into a wafer or disc with a 0.250-inch diameter and a thickness of 0.020 inch.

A constant program goal was to produce, by pressing on the F. J. Stokes rotary punch press, a gun propellant wafer having adequate strength to complete the ballistic cycle in an aircraft cannon without fracture. The immediate contractual goals were to achieve:

1. A composite solid propellant with an HMX oxidizer and a moldable binder.
2. A mass impetus not less than 360,000 ft-lbm (P_c = 5,000 psia).
3. An isochoric flame temperature not greater than 2,600°K (P_c = 5,000 psia).
4. A mean gas molecular weight of combustion products not greater than 20.00 Atomic Mass Units (P_c = 5,000 psia).

Later in the program an additional goal was specified. The burning rate exponent, n, should not exceed 0.90, in the log-log domain.

B. EQUIPMENT

Two rotary punch presses were employed to fabricate wafer gun propellant. The larger press has 41 punch and die stations, the smaller press has 16. Both machines were manufactured by the F. J. Stokes Company of Warminster, Pennsylvania. These presses were equipped with 0.250-inch-diameter flat face punch sets together with matched die plates, propellant hoppers, and guide shoes. Figure 1 illustrates the punch press operation, and Figure 2 shows the larger (41 station) F. J. Stokes press.

The K. R. Komarek Compactor (Figures 3 and 4) was used to compact milled propellant to a consistency which would flow through the F. J. Stokes press hopper until it was replaced functionally by the Dravo-Lurgi Disc, a 14-inch-diameter disc pelletizing device. Figure 5 illustrates this device.

HMX of large crystal size (Grade 2, Class C) was reduced to a weight mean diameter of 2 microns together with binder in a fluid energy mill. Figure 6 shows the operation of this device.

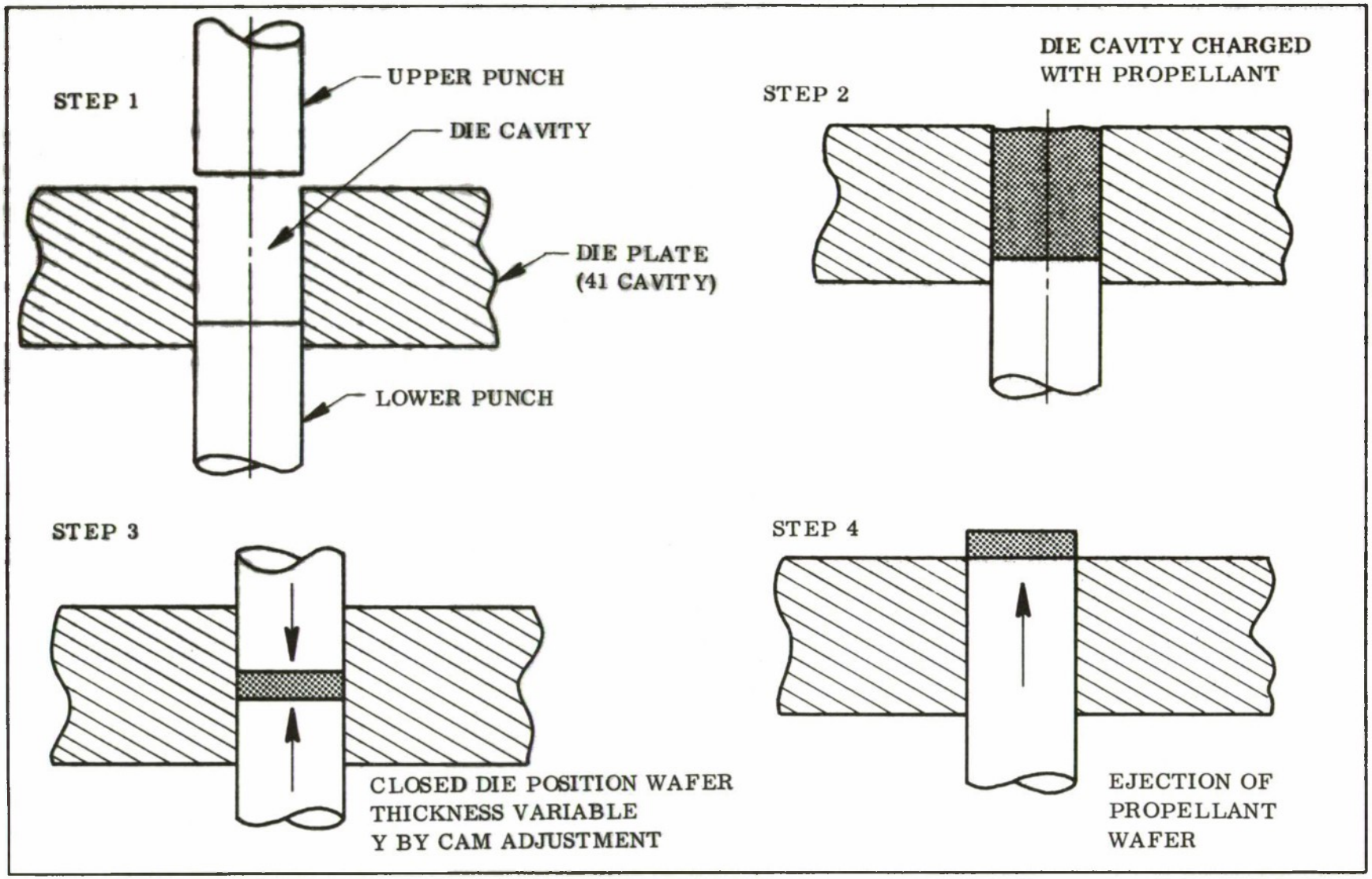

Figure 1. Operation Principle of F. J. Stokes Wafer Punch Press

Figure 2. F. J. Stokes Wafer Punch Press

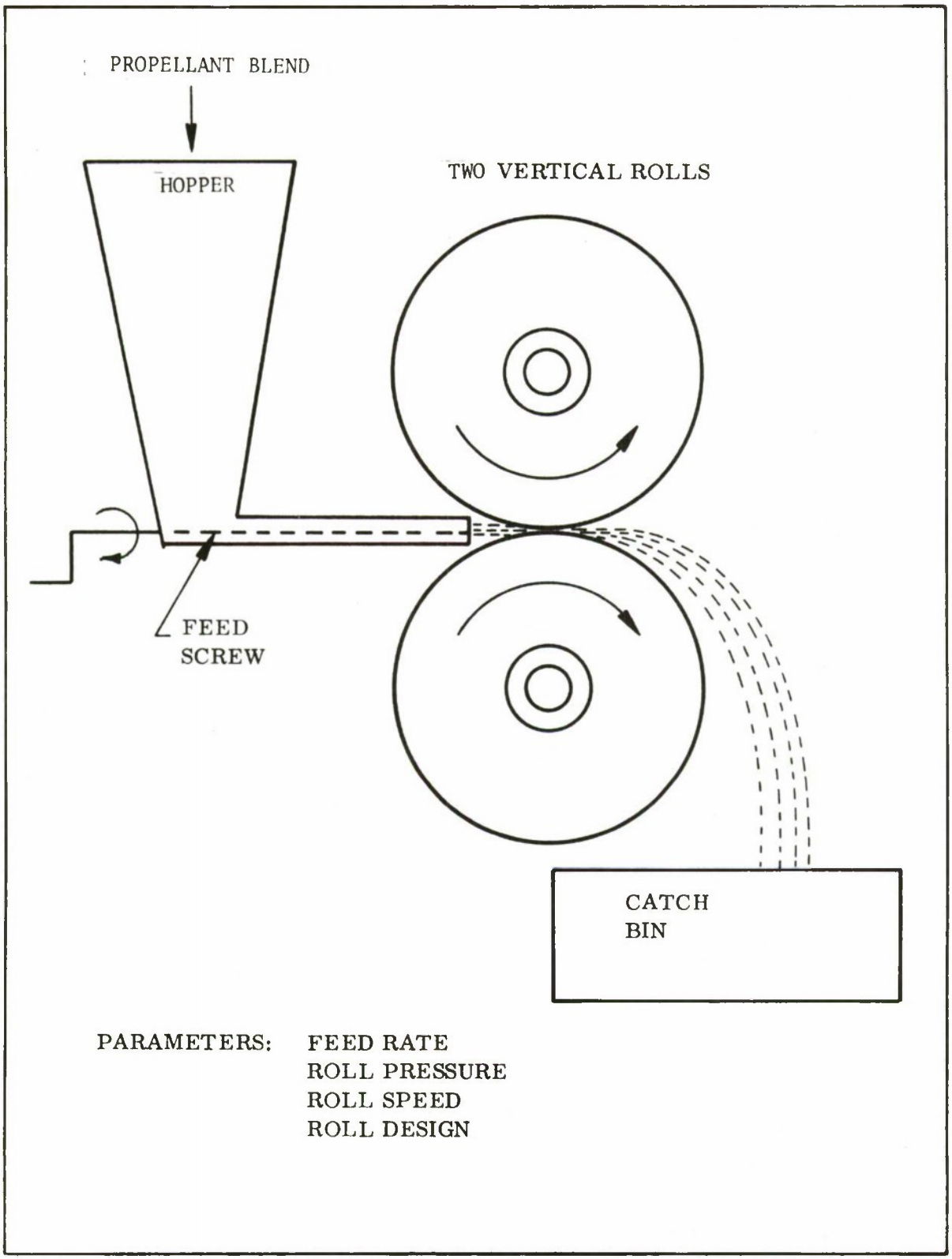

Figure 3. Diagram of K. R. Komarek Roll Compactor

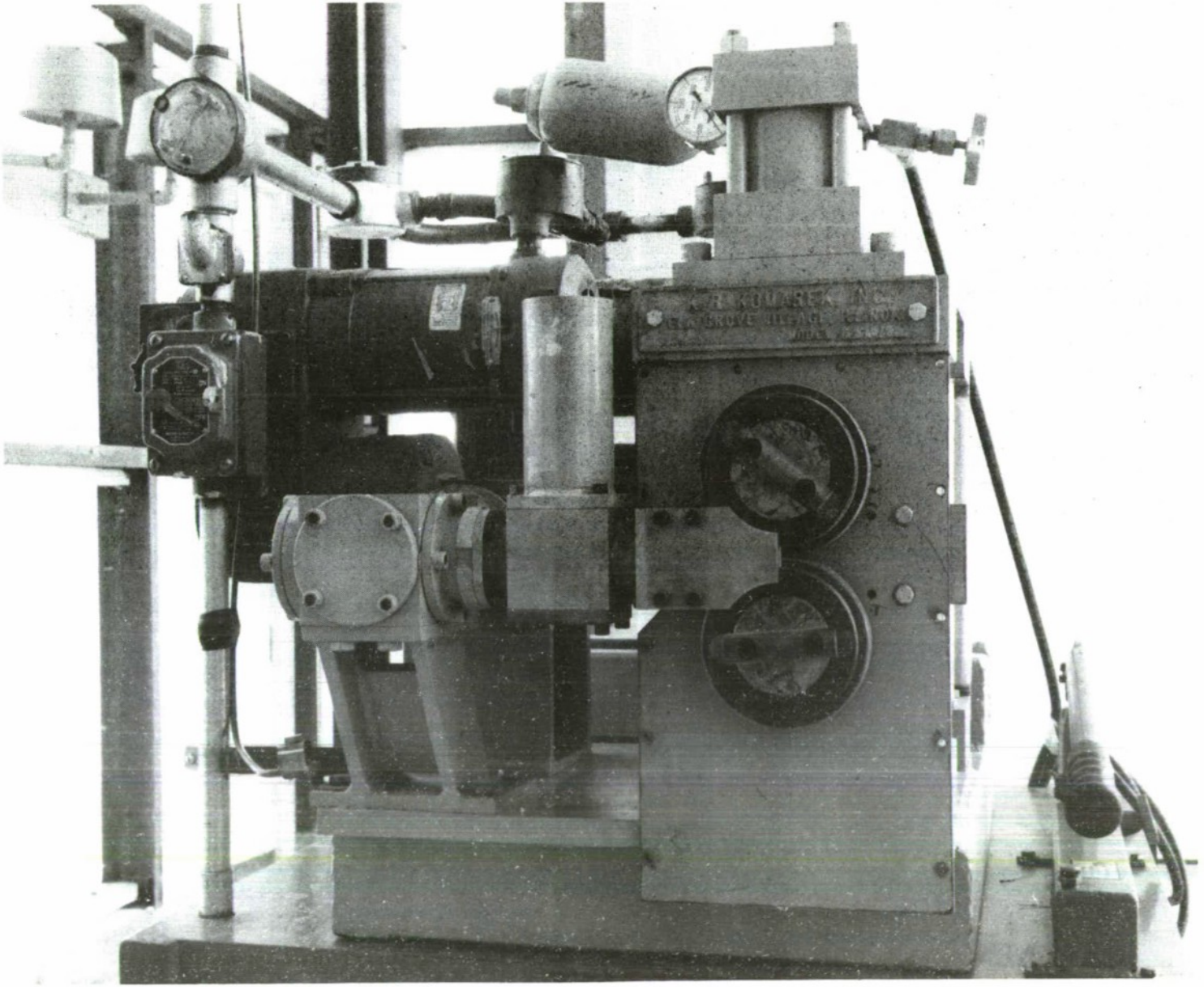

Figure 4. K. R. Komarek Roll Compactor

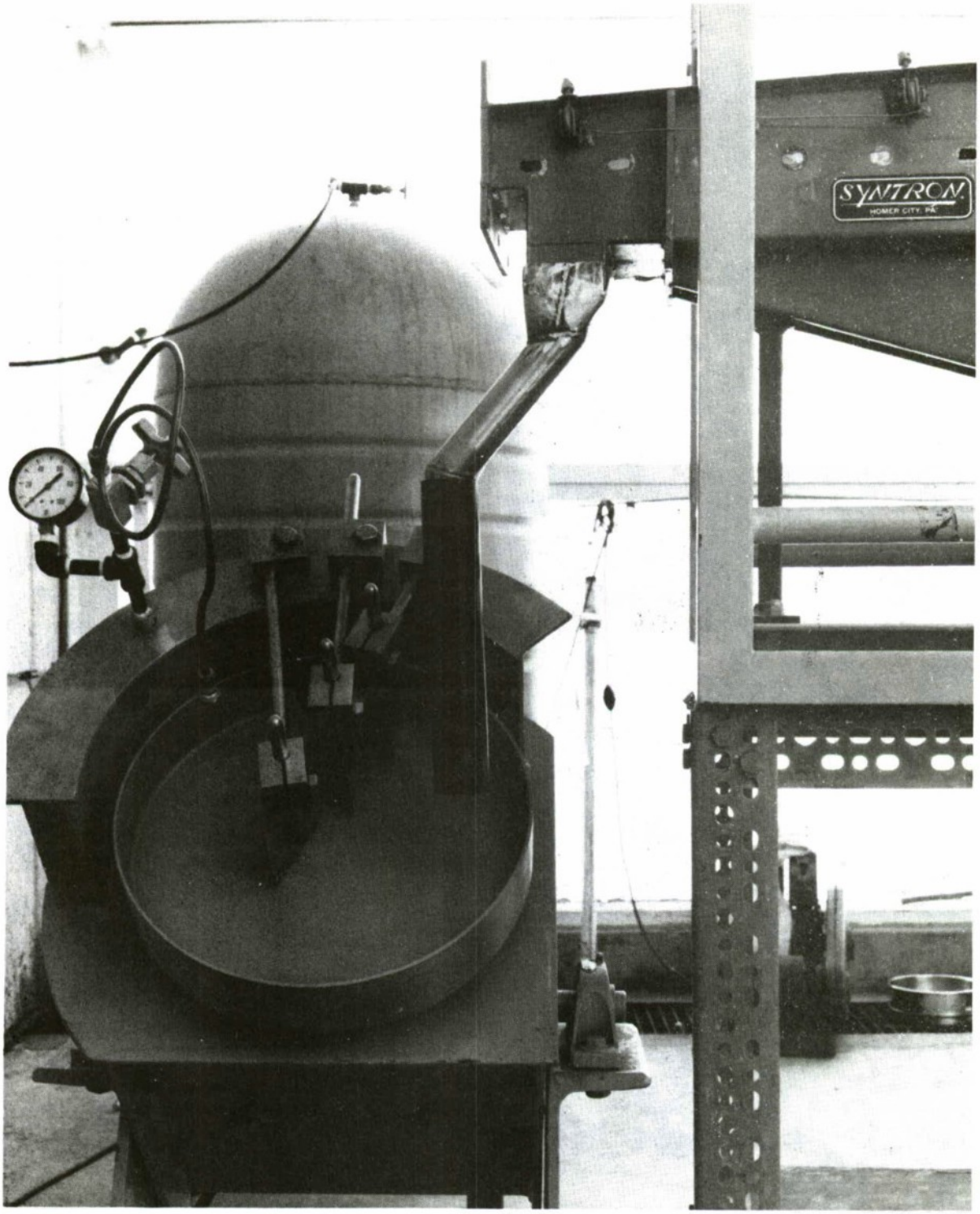

Figure 5. Dravo-Lurgi 14-Inch-Diameter Disc Pelletizer

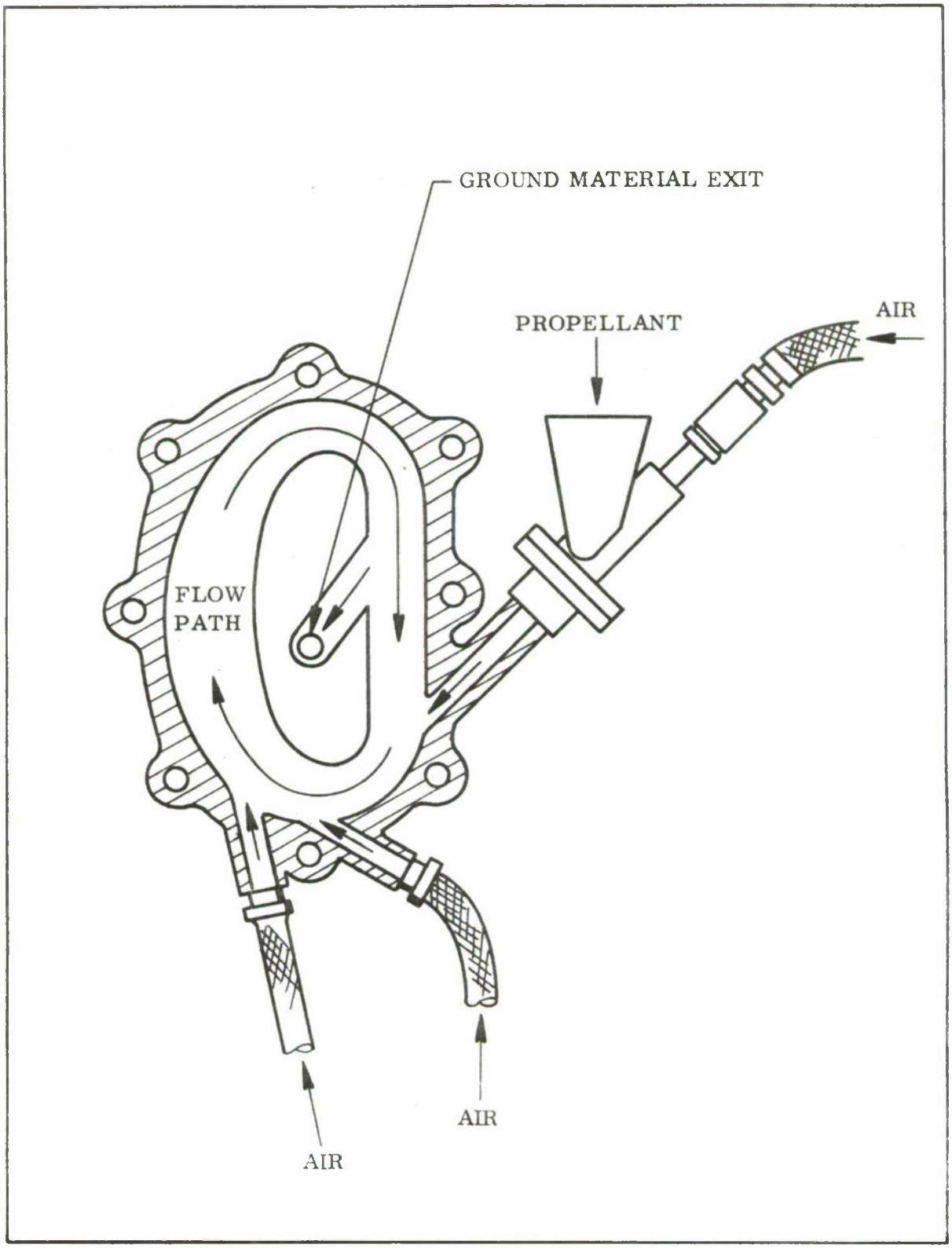

Figure 6. Diagram of Fluid Energy Mill

C. PROPELLANT THERMODYNAMIC CHARACTERISTICS

The mathematical model used to calculate theoretical isochoric flame temperature, mass impetus, and combustion gas molecular weight values was taken from NASA Technical Note TN D-1454 by Frank J. Zeleznik and Sanford Gordon. This model has been incorporated in Program S3096 at Thiokol Chemical Corporation/ Wasatch Division as the "H, S" program option. Table I lists the input information needed to calculate propellant performance. Impure HMX (assumes 1.0 percent RDX as an impurity) was used to calculate propellant performance. Actually, the HMX supplied by Holston Arsenal had an analyzed purity of 99.5 to 99.8 percent. Table II presents the final theoretical thermochemical characteristics for all propellant formulations studied in this program which could be processed into gun propellant wafers. The "Sample Code" letters of Table II and Table III are the same.

D. PROPELLANT FABRICATION PROCESS

All wafer gun propellant fabricated under this program was processed in the following pattern. Individual variations for particular propellant batches will be discussed later.

Process Sequence

1. Dry binder materials.
2. Screen all lumps from the binder materials.
3. Weigh the individual binder constituents.
4. Pre-blend binder constituents using the twin shell blender for small lots and the double cone blender for large lots.
5. Weigh Class C HMX.
6. Grind binder together with HMX in the fluid energy mill to 2-micron weight mean particle diameter.
7. Compact or agglomerate milled propellant:
    a. Compact with K. R. Komarek roll compactor, or
    b. Produce spheroidal agglomerates by solvent agglomeration or with the Dravo-Lurgi Disc pelletizer (14 inch).

TABLE I. CHARACTERISTICS OF PROPELLANT CONSTITUENTS

Material	Formula	Density (gm/cc)	Heat of Formation (K cal/gm)
HMX	$C_4H_8N_8O_8$	1.90	+0.0605
RDX	$C_3H_6N_6O_6$	1.80	+0.0661
Impure HMX	$C_{1.3506}$ $H_{2.7012}$ $N_{2.7012}$ $O_{2.7012}$	1.90	+0.0606
Isonate 136T (NCO)	$C_{16}H_{12}N_2O_2$	1.22	-1.199
C-4000 (DIOL)	$(C_2H_4O)_n$	1.20	-0.998
TMP (TRIOL)	$C_6H_{14}O_3$	1.16	--
Ferric Acetyl Acetonate	$FeC_{15}H_{21}O_6$	1.91	--
Solid Binder (NCO/DIOL/TRIOL) Ratio			
110/20/80	$C_{4.9617}H_{7.8950}N_{0.2132}O_{1.8413}$	1.239	-0.720
110/15/85	$C_{5.0406}H_{7.6793}N_{0.2532}O_{1.7607}$	1.245	-0.742
110/10/90	$C_{5.1554}H_{7.3654}N_{0.3115}O_{1.6433}$	1.250	-0.774

TABLE II. PROPELLANT THERMOCHEMICAL CHARACTERISTICS

Item No.	Sample Code	Percent HMX	Percent Graphite	Solid Binder NCO/DIOL/TRIOL	T_V, °K[a]	Impetus (ft-lbf/lbm)	Mean Molecular Weight of Products	Burning Rate r_b at 10,000 psia (in./sec)	Burning Rate[b] Exponent n
1	A[c], B	80.0	0	110/20/80	2,667	373,540	19.85	1.44	1.20
2	C, D	75.0	0	110/15/85	2,259	324,840	19.34	1.16	1.08
3	E, F	75.0	0	110/20/80	2,306	333,760	19.22	--	--
4	--	80.0	0	110/10/90	2,556	357,200	19.90	1.29	1.20
5	G, H, J, K	80.0	1.0	110/15/85	2,597	362,770	19.91	1.16	1.01
6	L, M, N	80.2	0.5	110/15/85	2,625	366,430	19.92	--	--
7	--	82.0	0	110/10/90	2,719	373,280	20.26	1.54	1.14
8	P, Q, R	82.0	0.5	110/15/85	2,766	379,640	20.26	--	--
9	--	82.0	1.0[d]	110/10/90	2,698	369,760	20.29	--	--
10	--	82.0	2.0[d]	110/10/90	2,676	366,180	20.33	--	--
11	--	82.0	3.0[d]	110/10/90	2,654	362,510	20.36	--	--
12	--	85.0	0	110/15/85	3,012	401,500	20.86	2.95	1.30

[a] P_c = 5,000 psia

[b] Burning rate equation: $r_b = A (P_{AVG}/10,000)^n$ in./sec; for Item No. 1 the equation is $r_b = 1.44 (P_{AVG}/10,000)^{1.20}$

[c] See Figure 7 for a photograph of Sample A

[d] See Figure 8 for a photograph of Item No. 9, 10 and 11

TABLE III. PHYSICAL CHARACTERISTICS OF GUN PROPELLANT WAFERS TESTED AT EGLIN AIR FORCE BASE

Sample Code	Batch No. RDLNB	HMX (percent)	Graphite (percent)	NCO/DIOL/ TRIOL Ratio	Nominal Thickness (in.)	Actual Thickness, avg (in.)	Density (gm/cc)	TMD[a] (percent)	Rupture Load (lbf)	Coating Material[b]	Film Thickness (in.)
A[d]	7297-010	80.0	--	110/20/80	0.030	0.031	1.62	95	4.2	--	--
B	7297-010A	80.0	--	110/20/80	0.020	0.022	1.57	92	1.7	--	--
C	7297-016A	75.0	--	110/15/85	0.030	0.037	1.56	94	4.4	--	--
D	7297-016A	75.0	--	110/15/85	0.020	0.029	1.54	93	3.4	--	--
E	7297-015A	75.0	--	110/20/80	0.030	0.033	1.58	95	5.0	--	--
F	7297-015A	75.0	--	110/20/80	0.020	0.022	1.57	95	1.6	--	--
G	F268-42A	80.0	1.0	110/15/85	0.021	0.0237	--	--	--	Porous acrylic ester film	0.001
H	F268-42B	80.0	1.0	110/15/85	0.021	0.0237	--	--	--	Solid acrylic ester film	0.001
J	F268-42C	80.0	1.0	110/15/85	0.021	0.0237	--	--	--	Sparse polyurethane film	0.001
K	F268-42	80.0	1.0	110/15/85	0.020	0.0227	--	--	--	None	0.000
L	F526-25A	80.2	0.5[c]	110/15/85	0.020	--	--	--	--	--	--
M	F526-25A	80.2	0.5[c]	110/15/85	0.025	--	--	--	--	--	--
N	F526-25A	80.2	0.5[c]	110/15/85	0.030	--	--	--	--	--	--
P	F526-25B	82.0	0.5[c]	110/15/85	0.020	--	--	--	--	--	--
Q	F526-25B	82.0	0.5[c]	110/15/85	0.025	--	--	--	--	--	--
R	F526-25B	82.0	0.5[c]	110/15/85	0.030	--	--	--	--	--	--

[a]TMD Theoretical maximum density

[b]Film coating on one side of wafer to limit initial burning surface.

[c]Graphite added at 0.5 grams per 100 grams after milling and before pressing propellant.

[d]See Figure 7 for a photograph of Sample A

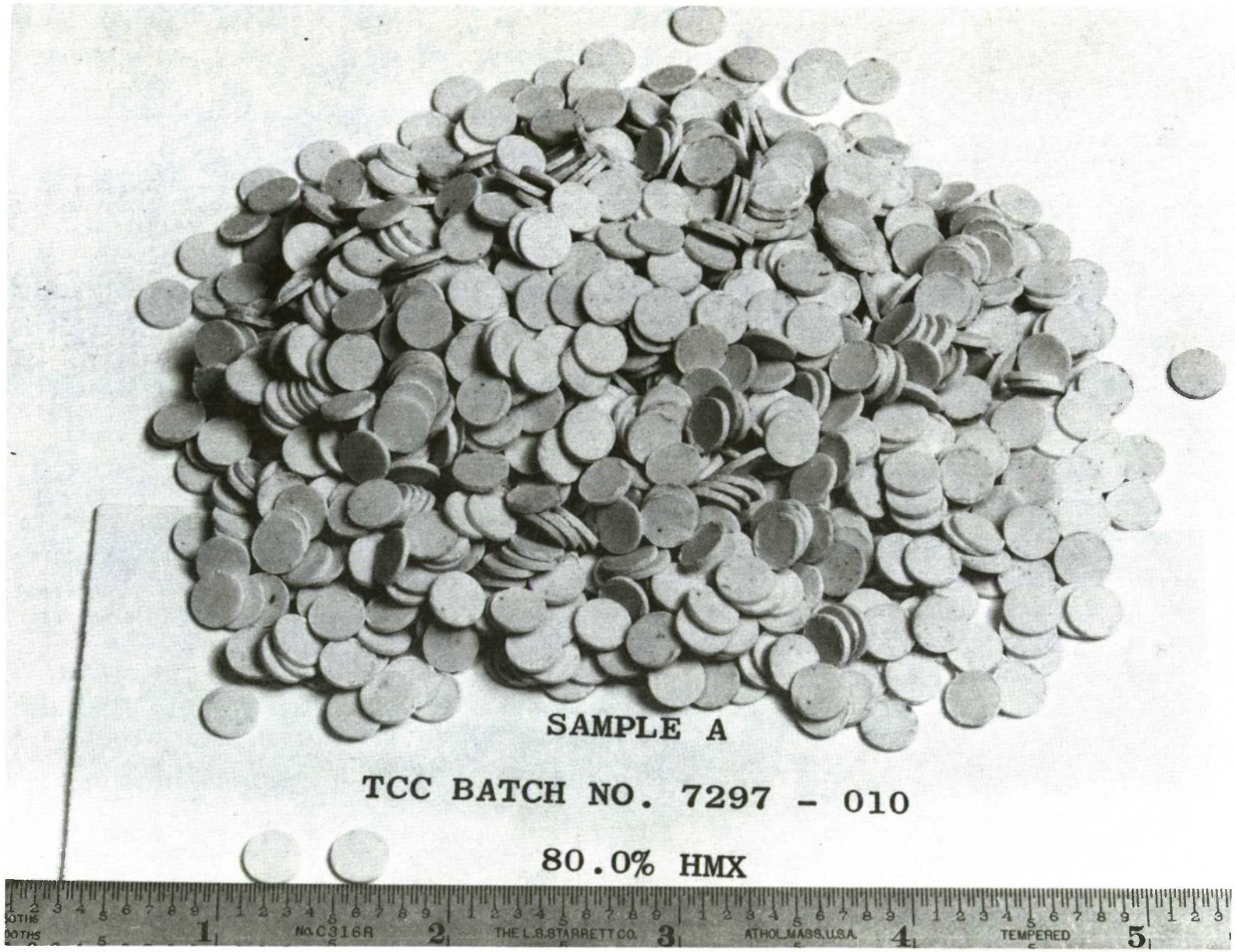

Figure 7. Sample A, TCC Batch No. RDLNB 7297-010, 80% HMX

82.0% HMX, 2 micron; Binder No. 110-10-90
1.0% Graphite, 0.020" thick
Pressed directly from 2 micron powder

82.0% HMX, 2 micron; Binder No. 110-10-90
3.0% Graphite, 0.030" thick
Repressed from #42 mesh material

82.0% HMX, 2 micron; Binder No. 110-10-90
3.0% Graphite, 0.020" thick
Repressed from #42 mesh material

82.0% HMX, 2 micron; Binder No. 110-10-90
3.0% Graphite, 0.020" thick
Pressed directly from 2 micron powder

Figure 8. Wafer Gun Propellant Samples Pressed With Graphite

8. Process compacted propellant through a Hobart Mixer using a No. 30 U. S. Standard screen. (Omit step 8 if step 7b is elected and go to step 9.)

9. Press propellant wafers on the F.J. Stokes pelleting punch press at room temperature.

10. Oven cure propellant wafers at 210° F for 2 hours (plus or minus 1/2 hour).

E. PROPELLANTS EVALUATED

Initially all samples of wafer gun propellant contained 82.0 percent HMX and 18.0 percent of a polyethylene glycol/polyurethane dry binder, NCO/DIOL/TRIOL ratio of 110/15/85.* The crystalline oxidizer, HMX, was milled to a weight mean diameter of 2 microns by means of a fluid energy mill in every case. It is fundamental to the production of a gun propellant containing HMX that the crystal size of the HMX be held to a minimum in order that the burning rate exponent, n, may also be a minimum. Later in the program, propellant samples were fabricated containing a fluidizer (e.g., Alon G) or a lubricant (graphite).

Difficulty was encountered in causing the light and fluffy blend of 2-micron HMX and binder to flow through the feed hopper of the F. J. Stokes press. HMX has an angle of repose which causes bridging in the feed hopper, thus blocking flow to the press platen. Also, the 2-micron propellant powder has a strong tendency to cure to the press punches and die cavity. This is hazardous as HMX is friction sensitive (Table IV). Accordingly, means were investigated to enhance propellant flow properties in the F. J. Stokes press.

Difficulty was also experienced in pressing wafers as thin as 0.020 inch due to a punch press design problem. The press was altered to permit pressing the specified wafer thickness.

In order to enhance propellant flow it was determined to compact propellant with the K. R. Komarek roll compactor (Figures 3 and 4) prior to pressing. This compactor produces platelets of propellant which must be screened with a No. 30 U. S. standard screen mesh before pressing is done. Propellant which had been roll compacted did flow well through the F. J. Stokes press but was unsatisfactory in two respects. First, the propellant would pre-cure in the roll compactor, sticking to the rolls and creating a hazardous condition. Secondly, when oven curing the pressed wafers, the pre-cured grains would not bond together well to form a tough propellant wafer. Consequently, the final product was friable and would break up during the ballistic cycle of a gun firing. It should be noted here that all 82.0 percent HMX propellant samples contained the binder cure catalyst ferric acetyl acetonate, $Fe(AA)_3$.

Fluidizers at the 0.5 percent level were added to propellant samples containing 82.0 percent HMX in order to reduce the pre-cure tendency in the roll compactor. Fluidizers used were MoS_2, Alon G (Al_2O_3), Aerosil R972 (SiO_2), and Teflon.

In addition, a lubricant, graphite, was also incorporated into 82.0 percent HMX propellant at the following percent levels: 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0. This was expected to reduce pre-curing. Neither the fluidizer nor the lubricant

*All percents mentioned in this report are weight percents.

TABLE IV. PROPELLANT HAZARD CHARACTERISTICS

	Propellant	Electrostatic Sensitivity, joules	50% Impact Sensitivity, inches	50% Friction Sensitivity, pounds	Autoignition Time, hours (300°F)	Autoignition Time, seconds (550°F/450°F)	Flash Point, °F
1.	75.00% HMX[a] Binder L-35	>8	>46	>64	>24	20/300	525
2.	80.00% HMX Binder 110/10/90	>8	35	>64	>24	--/284	--
3.	85.00% HMX Binder 110/15/85	>8	35	>64	>24	18.5/200	518
4.	IMR 4064 Control	--	--	--	--	2.6/7.2	356

[a] Binder L-35 (NCO/DIOL/TRIOL ratio of 110/15/85 is the extrudable equivalent of the dry binder system in Item 3 above.

TABLE V. PROPELLANT THERMAL STABILITY

	Propellant	Differential Thermal Analysis, °F (9°C/minute)	Thermal Gravimetric Analysis, °F (12°C/minute)	DTA, TGA and AIT Product	Explosion Temperature Test (5 seconds), °F	Cookoff Time (550°F), seconds (brass cased)
1.	75.00% HMX[a] Binder L-35	480	509-518	Smoke	664	>20
2.	80.00% HMX[b] Binder 110/10/90	470	--	Smoke	666	--
3.	85.00% HMX[c] Binder 110/15/85	480	482	Smoke	664	20
4.	IMR 4064[d] Control	370	--	Flame	469	8

[a] Binder L-35 (NCO/DIOL/TRIOL ratio of 110/15/85 is the extrudable equivalent of the dry binder system in Item 3 above.

[b] Propellant used is the same as Item No. 4 in Table II on page 14.

[c] Propellant consisted of 85.0 weight percent of 2-micron HMX and the solid polyurethane binder with an NCO/DIOL/TRIOL ratio of 110/15/85.

[d] IMR 4064 is a standard DuPont product.

prevented pre-curing and the lubricant added at levels above 1.0 percent produced considerable carbon fouling in test guns. Therefore, the use of roll compaction and fluidizers was discontinued.

When fluidizers were introduced in an effort to make a harder and less friable wafer, the binder NCO/DIOL/TRIOL of 110/15/85 was changed to 110/10/90. However, making the wafers harder did not increase wafer toughness.

Solvent agglomeration of blended 2-micron propellant was investigated by the Explosive and Ordnance Development Section as a substitute for roll compaction to provide spheroids of propellant which would flow well in the F. J. Stokes press. Five common solvents were studied:

1. Trichloroethane
2. Methyl ethyl ketone (MEK)
3. Dimethyl ketone (acetone)
4. Dichloroethane (methylene chloride)
5. Methyl alcohol (methanol)

Two propellant formulations were agglomerated, 80.2 and 82.0 percent HMX with the 110/15/85 binder. Following agglomeration all solvent was removed by air drying at ambient temperature and vacuum drying at 165°F. Graphite, at the 0.5 percent level, was added to the agglomerated spheroids before pressing to enhance flow properties and to minimize the amount of graphite required. Six propellant samples were provided to Eglin Air Force Base for cannon testing.

Samples

	(1)	(2)	(3)	(4)	(5)	(6)
Percent HMX	80.2	80.2	80.2	82.0	82.0	82.0
Diameter (in.)	0.250	0.250	0.250	0.250	0.250	0.250
Thickness (in.)	0.020	0.025	0.030	0.020	0.025	0.030
Percent Graphite	0.5	0.5	0.5	0.5	0.5	0.5

Test firing these samples showed wafer break-up in the cannon and pressure excursions which are associated with grain break-up. Graphite added after agglomeration appears to aggravate wafer break-up.

A propellant batch consisting of 80.0 percent HMX, 1.0 percent graphite milled into the propellant at the 2-micron level, and 19.0 percent binder with NCO/DIOL/TRIOL ratio of 110/15/85 was prepared. The theoretical performance of this propellant meets contract specifications except for the burning rate exponent (Table II). The use of graphite was continued as a safety measure for pressing purposes.

Four propellant samples were prepared for cannon testing at Eglin Air Force Base. Three of the four samples were coated on one side of the wafer to deter burning. These propellant samples had the following characteristics.

Propellant Specifications

HMX (2-micron)	80.0%
Graphite (2-micron)	1.0%
Binder (NCO/DIOL/TRIOL: 110/15/85)	19.0%
Catalyst: $Fe(AA)_3$	0.20 gram per 100 grams of propellant

Sample No.	Diameter (inches)	Average Thickness (inches)	Coating Thickness (inches)	Coating Material
1[a]	0.250	0.0237	0.001	uniform acrylic ester film
2[a]	0.250	0.0237	0.001	porous acrylic ester film
3[a]	0.250	0.0237	0.001	sparse polyurethane film
4[a]	0.250	0.0227	none	no film

The equation of burning for propellant sample No. 4, above, is given in Item No. 5 of Table II. When these samples were fired in the 30/20mm cannon at Eglin Air Force Base, wafer break-up was noted. Accordingly, studies were undertaken to improve the physical properties of the propellant wafers. Previously, laboratory-prepared samples had demonstrated excellent physical strength; however, manufactured wafers did not possess the same physical strength. The effects

[a]Samples No. 1,2, and 3 were prepared by coating Sample No. 4.

of solvent agglomeration, heat agglomeration, and graphite addition on the physical strength of pressed wafers are illustrated in Table VI. Specimens reported in this table were from the following propellant sample.

Propellant Composition

	%
HMX (2-micron)	80.00
Isonate 136T	8.15
Carbowax C-4000	9.40
Trimethylolpropane (TMP)	2.25
$Fe(AA)_3$	0.20
NCO/DIOL/TRIOL Ratio	110/10/90

Wafers 0.500 inch in diameter and 0.020-inch thick were pressed from this material and cured at 210° F for 2.0 hours. The wafer-breaking strength or friability was determined by centrally loading the wafer with a 0.100-inch-diameter ram which was connected to a sensitive load cell. The wafer perimeter was supported. Table VI data illustrate the comparative strengths of the wafers produced. When no solvent or pre-curing condition was applied to the composition prior to pressing, the best physical strength was obtained. Pre-curing the binder gives strength comparable to an uncured binder system. Solvent agglomeration treatment of the material gives strengths less than half that of the unagglomerated material. A poor solvent such as Freon 113 gave higher strengths than good solvents like methylene chloride.

These tests demonstrated that:

1. Solvents effectively pre-cure the binder system by allowing intimate contact of binder constituents.

2. Pressing without pre-curing produces wafers twice as strong as the pre-cured wafers.

3. Addition of 1.0 percent graphite to nonagglomerated fluid energy milled propellant prior to pressing increases wafer strength.

A methodology was required which would permit a propellant composition to be solvent agglomerated without pre-curing. Isocyanate blocking agents and cure catalyst levels were investigated to determine their effectiveness in preventing pre-curing conditions.

TABLE VI. PROPELLANT PROCESSING VS RUPTURE LOAD[a]

Sample	Pre-pressing Processing[b]	Cure at 210°F (hours)	Rupture Load[c] (lbf)
1	None	2.0	1.3
2	1.0% graphite	2.0	1.5
3	None	None	0.6
4	Pre-cure[d] 2.0 hours	2.0	0.4
5	Pre-cure[d] 2.0 hours 1.0% graphite	2.0	0.5
6	Pre-cure[d] 0.25 hour	2.0	0.6
7	Pre-cure[d] 0.25 hour 1.0% graphite	2.0	0.6
8	Methylene chloride agglomerated	2.0	0.3
9	Methanol agglomerated	2.0	0.6
10	Freon 113 agglomerated	2.0	0.8

[a] Propellant Composition: 80.0% HMX, 20.0% Binder 110/10/90, no graphite and no fluidizer.

[b] Pressing pressure above 50 kpsi.

[c] Force required to rupture 0.50 inch diameter by 0.020 inch thick disc with a 0.10 inch ram.

[d] Pre-cure temperature was 210°F.

Three isocyanate blocking agents (acetylacetone, ethyl acetoacetate, and azobisisobutyronitrile) were studied. Two ferric acetyl acetonate catalyst levels, 0 and 0.20 percent, were investigated in a wafer gun propellant binder gum stock ratio of 110/10/90 (NCO/DIOL/TRIOL). The blocking agents were added to the blended binder system in an amount equal to the NCO equivalents. The gum stocks containing each blocking agent on both catalyst levels were divided into two parts. One part was dissolved in methylene chloride. Both of these samples were allowed to stand at 80° F for 24 hours allowing the methylene chloride to evaporate from the sample. The condition of each sample was observed at the 24-hour point; then the samples were oven cured at 210° F for 2 hours. The status of the cured binder samples is tabulated in Table VII.

Those binder samples which contained no cured catalyst showed no pre-curing when methylene chloride was the solvent. These samples oven cured to a tough rubbery consistency. The samples which contained cure catalyst all showed some tendency to pre-cure when treated with methylene chloride. Therefore, the most effective way to prevent pre-curing is to eliminate the cure catalyst, $Fe(AA)_3$. With the cure catalyst eliminated, the isocyanate blocking agents are not required to prevent pre-curing.

A 2-kg batch of wafer gun propellant was prepared at 80.0 percent HMX (2-micron) and 20.0 percent of binder, ratio of 110/10/90, NCO/DIOL/TRIOL, without cure catalyst or graphite. This batch was fluid energy milled, solvent agglomerated with methylene chloride, and pressed into 0.250-inch-diameter by 0.020-inch-thick wafers on the F. J. Stokes punch press. After each step, 0.500-inch-diameter by 0.020-inch-thick wafers were pressed from the same material, and the wafer rupture was load tested. The rupture load for these conditions is presented below.

0.500-Inch-Diameter Wafer

	Fluid Energy Milled	Solvent Agglomerated	F. J. Stokes Pressed
Rupture Load (lbf)	1.9	1.8	1.8

The strength of the no-catalyst wafers pressed on the F. J. Stokes press was compared with that of the four sample batch of wafers submitted to Eglin Air Force Base for test and evaluation (Samples Codes G, H, J and K in Table II).

TABLE VII. BLOCKING AGENT AND CATALYST LEVEL STUDY

	Blocking Agent	$Fe(AA)_3$ Catalyst Level (%)	No Solvent		Methylene Chloride (air dried)	
			Uncured	Cured	Uncured	Cured
1.	None	0	Crystalline	Cured tacky	Wax	Tough rubber
2.	Acetyl acetone	0	Wax	Cured tacky	Wax	Tough rubber
3.	Ethyl acetoacetate	0	Wax	Cured tacky	Wax	Tough rubber
4.	Azobisisobutyronitrile	0	Wax	Cured foam	Wax	Cured foam
5.	None	0.2	Cured	Cured	Cured	Cured
6.	Acetyl acetone	0.2	Damp wax	Cured	Liquid	Cured
7.	Ethyl acetoacetate	0.2	Cured crumb	Cured crumb	Thick syrup	Cured
8.	Azobisisobutyronitrile	0.2	Crystalline	Cured crumb	Cured	Cured

0.250-Inch-Diameter Wafers

	No Catalyst Formulation	Formulation Submitted to Eglin
Rupture Load (lbf)	2.2	2.3
Average Thickness (in.)	0.017	0.0227[a]
Percent HMX (2-micron)	80.0	80.0
Percent Graphite	None	1.0
Percent Theoretical Density	92.0	93.0
Relative Strength (%)	236.9	100.0
NCO/DIOL/TRIOL Ratio	110/10/90	110/15/85

The elimination of pre-curing resulted in a 136.9 percent increase in pellet strength even though no graphite was present in the improved wafer. However, these new wafers remained friable enough to shatter upon ignition in a cannon. In the calculation of relative strength of the 0.250-inch-diameter wafers reported above, allowance was made for the difference in thickness between the wafers. The relative strength values represent a normalized value with the strength contribution due to thickness factored out.

In order to decrease wafer friability two approaches were investigated. Effort was directed at increasing propellant bulk density by improving propellant solvent agglomeration and by changing the binder NCO/DIOL/TRIOL ratio from 110/10/90 to 110/20/80. A reduction of the binder crosslink density was expected to give a flexible, nonfriable wafer. A batch of propellant with a NCO/DIOL/TRIOL ratio of 110/20/80, 80.0 percent HMX (2-micron) was prepared. The batch was fluid energy milled and solvent agglomerated. The 0.500-inch-diameter wafers were pressed to evaluate formulation strength. A rupture load of 1.0 lb at a wafer density of 1.61 gm/cc was achieved. This is approximately one half the strength of the 110/10/90 equivalent formulation. However, the wafer rupture pattern was much different. The wafers did not shatter, but a hole was punched through the wafer.

[a]This propellant sample was identical to Sample No. 4 on page 23. Samples No. 1, 2, and 3 were prepared by coating Sample No. 4.

This indicated that the propellant had a tearing tendency. These wafers, upon handling, appeared far more flexible than previous formulations.

A burning rate change was noted between the 110/10/90 and 110/20/80 binder formulas (see Figure 9). The 110/20/80 formula burned approximately 12 percent faster at 10,000 psia (Table II, items 1 and 4). This may be due to the 110/20/80 formulation having a higher density, 1.62 gm/cc, compared with 1.57 gm/cc for the 110/10/90 formulation. A higher density product would have fewer micro-voids and therefore more continuity (a shorter molecular "mean-free-path") as seen by the traveling flame front. Burning rate exponents were identical at $\eta = 1.20$ and there were no slope-breaks in the pressure-regression rate plot from 3,000 to 15,000 psia average pressure.

A new method of solvent agglomeration was instituted which permitted agglomeration with methylene chloride and yielded a narrow particle size range. This method entailed using a Dravo-Lurgi Disc pelletizer to enlarge solvent-wetted clumps of material into porous spheres which feed very well into the F. J. Stokes press (Figure 5). No HMX crystal growth has ever been observed with methylene chloride solvent agglomeration.

In order to increase wafer density, a change in presses was studied. The 16-station F. J. Stokes rotary press was substituted for the 41-station F. J. Stokes rotary press previously used. The 41-station press is characterized by a dwell time of about 25 milliseconds while the 16-station press has a dwell time 8.75 times longer or about 219 milliseconds. This longer dwell time was expected to give better wafer compaction. Both presses were operated at a pressure of 100,000 psi at the end of the press punch stroke. A batch of 80.0 percent HMX gun propellant was fluid energy milled, Dravo-Lurgi Disc agglomerated, and pressed into 0.250-inch-diameter wafers on the 16-station press. This propellant had the following characteristics:

1.	HMX (2-micron) %	80.0
2.	NCO/DIOL/TRIOL ratio	110/20/80
3.	Thickness, inches (Average)	0.031
4.	Density, gm/cc	1.62
5.	Rupture Load, lbf	4.2
6.	Percent Theoretical Density	95
7.	Relative Strength, %	75

Although the rupture strength was nearly twice that of previous 0.250 inch diameter pellets, the relative strength, allowing for the strength contribution due to thickness, was 75 percent of the original 80.0 percent HMX formulation with $Fe(AA)_3$ and binder equivalents of 110/10/90. However, these wafers did not shatter when ruptured but fractured in half.

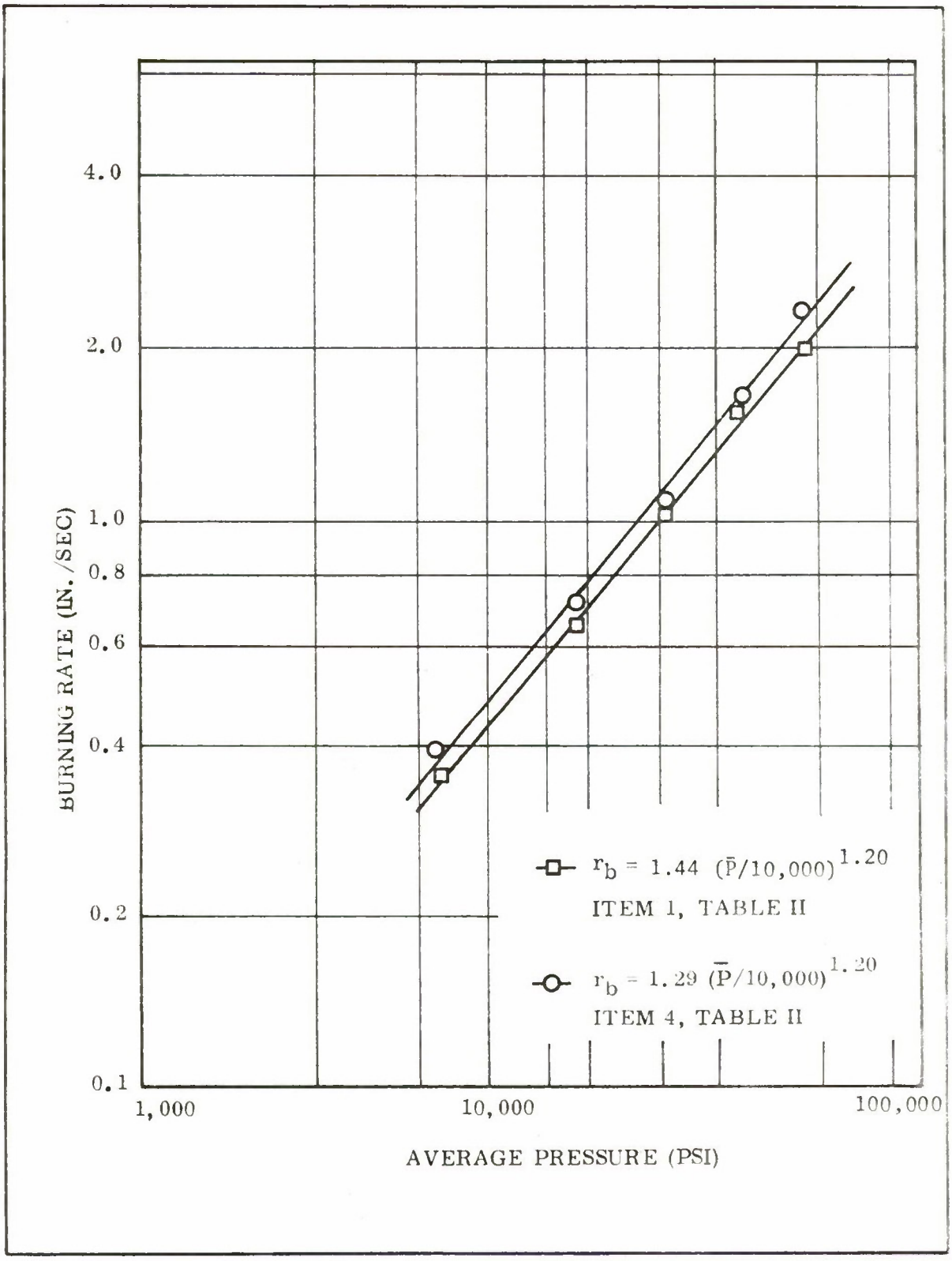

Figure 9. Burning Rate vs Pressure Curve for 80% HMX (2-micron)

Since the 80.0 percent HMX formulation with binder equivalents at 110/20/80 produced a fracture pattern which appeared to be an improvement over propellant with lower DIOL/TRIOL ratios, a sample was prepared for cannon testing at Eglin Air Force Base. Since wafer fracture was a problem, binder concentrations up to 25.0 weight percent were evaluated. The following compositions were supplied for government testing:

Sample Code	Percent HMX	NCO/DIOL/TRIOL Ratio	Nominal Thickness (in.)	Actual Average Thickness (in.)
A	80.0	110/20/80	0.030	0.031
B	80.0	110/20/80	0.020	0.022
C	75.0[a]	110/15/85	0.030	0.037
D	75.0[a]	110/15/85	0.020	0.029
E	75.0	110/20/80	0.030	0.033
F	75.0	110/20/80	0.020	0.022

As a result of gun testing, none of the above propellant formulations were selected for production as a final formulation, since all six samples had a low inherent loading density. It was noted, however, that the 75.0-percent HMX samples gave smooth regular pressure-time traces in the cannon, and the wafers which contained binder ratios of 110/20/80 with 75.0-percent HMX did not fracture upon ignition. Because of the low burning rates, all samples had web thicknesses too large to permit consumption of the propellant during its residence time in the cannon.

[a] For additional data, see Figure 10 and Table VIII.

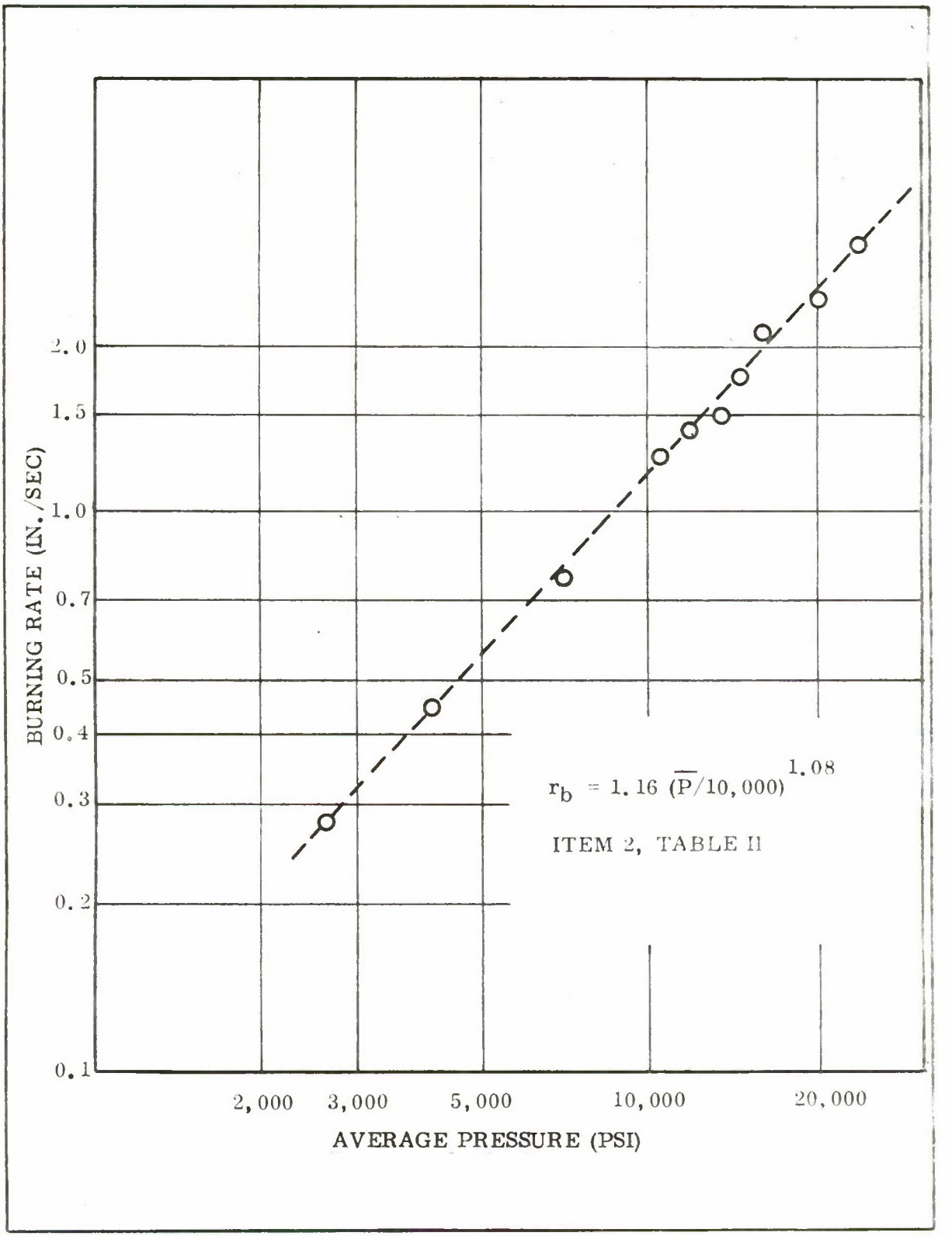

Figure 10. Burning Rate vs Pressure Curve for 75% HMX (2-micron)

TABLE VIII. IMPULSE BOMB DATA FOR 75.0% HMX (2-MICRON)[a]

Charge (gm)	Loading Density (Δ) (gm/cc)	Burn Time (msec)	Burning Rate (in./sec)	Pressure (psi) Max.	Pressure (psi) Avg	Experimental Impetus (F) (ft-lbf/lbm)
1.50	0.046	53.0	0.28	5,250	2,625	251,187
2.26	0.069	33.0	0.45	8,100	4,050	252,135
3.65	0.111	19.5	0.77	14,000	7,000	258,675
4.81	0.147	13.8	1.10	20,000	10,000	267,738
5.00	0.153	12.0	1.25	21,000	10,500	268,200
5.50	0.168	10.7	1.40	23,500	11,750	268,491
6.01	0.183	10.0	1.50	26,500	13,250	272,938
6.40	0.195	8.5	1.76	29,250	14,625	278,570
7.15	0.218	7.5	2.00	32,000	16,000	264,818
8.30	0.253	6.8	2.22	40,000	20,000	272,463
9.45	0.288	5.0	3.00	48,000	24,000	273,764

[a] Burning rate equation $R_b = 1.16\ (\overline{P}/10,000)^{1.08}$
Same formulation as Item No. 2, Table II.

SECTION V

CONCLUSIONS

Wafer gun propellant formulations (HMX content from 75.0 to 82.0 percent) developed on this program with 0.250-inch diameter and thicknesses from 0.017 inch to 0.037 inch have inadequate packing density for ballistic usefulness when fired in a 30mm GAU-8/A cannon.

The concept of pressing gun propellant wafers is simple and direct. However, it is a slow process on present equipment. Larger and faster tools are required for volume production.

Punch pressing is a useful technique for processing gun propellant which features a dry binder system. Dry binder systems at present permit a higher weight percent loading of ballistic solids than do liquid binder systems.

Further improvements are possible in the processing technique. The use of Dravo-Lurgi Disc agglomerated propellant as a feed material for the F. J. Stokes press solved feed problems. Removal of cure catalyst from the binder solved pre-curing difficulties. Greater wafer density is possible with additional study.

Wafer gun propellant friability (relative crush strength) is a function of binder selection, binder cure, wafer density, and the ratio of thickness to diameter. A wafer 0.125 inch in diameter and 0.020-inch thick would appear much stronger than the 0.250-inch-diameter by 0.020-inch-thick wafers which were the subject of this program.

Wafer gun propellant packing density in a cartridge case is a function of wafer density, the ratio of wafer thickness to diameter, and the ratio of wafer diameter to case diameter. As wafer diameter decreases, packing density increases for a constant cartridge case internal diameter and volume.

When HMX is the nitramine of choice for a gun propellant, slope-break in the burning rate versus average pressure function (for constant volume) may be avoided by using an oxidizer weight mean crystal diameter of 2 microns or less. It is also helpful to employ not more than 75.0 percent HMX in the absence of an HMX ballistic modifier designed to lower the burning rate exponent, n, as there is a quantity effect upon the magnitude of n as well as an ozidizer particle size effect (ref Figures 9 and 10).

SECTION VI

RECOMMENDATIONS

Wafer gun propellant fabrication should be investigated by cutting discs, squares, or diamonds from thin sheets of roll calendered or extruded propellant. Dry binders may be roll compacted or solvent extended for sheet extrusion.

HMX has a relatively low burning rate without a ballistic modifier in the 2-micron weight mean diameter. The production of wafers of 0.005 to 0.010 inch web thicknesses (to obtain short burning time) in diameters small enough to permit a high packing density in existing cartridge cases should be studied.

Burning rate modifiers to be used with a cyclic nitramine and a dry or liquid binder system to further increase burning rate and lower burning rate exponent should be studied.

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13. ABSTRACT

The wafer gun propellant studied in this program is a composite solid propellant with a moldable binder. It contains the cyclic nitramine, HMX, for an oxidizer and has the advantages of high impetus, relatively low flame temperature, and a manufacturing process adaptable to high volume production with good producibility. The wafer gun propellant is punch-pressed from a dry powder into a disc or wafer with nearly neutral burning characteristics. It is then oven cured. The wafer configuration permits an easily varied burning web, since the pressed wafer thickness is a variable controlled by changing pressing dies. The pressed wafer fabrication technique permits a higher oxidizer solids loading than is presently possible with a conventional extruded composite propellant. Production involves fewer process steps than a conventional colloidal gun propellant.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Wafer Gun Propellant						
Wafer Gun						
Colloidal Gun Propellant						
Composite Solid Propellant						
Propellant Isochoric Flame Temperature						
Wafer Gun Propellant Fabrication						
Pressable Composite Solid Propellant Formulation						